A Thesis on

Reactive Extraction of Acetic Acid

Submitted by

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CERTIFICATE

This is to certify that the thesis titled "*Reactive Extraction of Acetic Acid*" Submitted to the National Institute of Technology Rourkela by TarunVerma, Roll no.212CH1082, for the award of the degree of master of Technology in chemical engineering. This is a bonafide record of research work carried out by him under my supervision and guidance. The candidate has fulfilled the entire prescribed requirement. The thesis, which is based on candidate's own work, has not been submitted elsewhere for a degree.

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ABSTRACT

Acetic acid is an important carboxylic acid widely used in chemical, pharmaceutical, food and other industries. The growing importance of biological production (fermentation) expressed with new routes and increasing production rates, leads to look for technologies of downstream processing for product separation. The reactive extraction can be used for the recovery of an acetic acid from aqueous solution.The reactive extraction with specific extractant and a proper combination of extractant and diluents provides the higher capacity and selectivity of an acetic acid. The recovery of acetic acid by reactive extraction is studied using an organic solvent such as toluene, petroleum ether and n-hexane. The feed concentration of acetic acid in the aqueous solution ranges in between 0.1 to 0.4 gmol/l.

The extraction of acetic acid from aqueous solution (feed concentration 0.4 gmol/lit) with organic solvent gives the distribution coefficient (K_D) in toluene, petroleum ether and N-Hexane solvents equal to 1.29, 0.79 and 0.6 respectively. Further, the reactive extraction is carried out with the mixtures of extractant–diluents. The tri-iso-octylamine is used as an extractant. The extraction of acetic acid with 40% of tri-iso-octylamine in an organic solvent shows the highest distribution coefficient for toluene, petroleum ether and n-hexane. These are 3.65, 2.70 and 2.63 respectively. By comparing results, it can be concluded that using the mixtures of extractant–diluents, the extraction is significantly improved.

Keywords: Acetic acid, Reactive extraction, TIOA, Diluents, Equilibrium, tri-iso-toctylamine

CHAPTER 1

INTRODUCTION

1 Introduction

1.1 Reactive Extraction

Reactive extraction is a process intensification method coming under reactive separations and is gaining great attraction particularly in terms of its comparison with the conventional extraction method, which have posed a great economic pressure to the industries carrying out them. Reactive extraction links chemical sources and sinks to enhance reaction rates, conversions, and selectivity. Most of the chemical processes are equilibrium driving, and removal of product as soon as it is producewould lead to enhanced reaction rates.

For last few decades, there have been intensive works on the reactive extraction for the recovery of carboxylic acid, metals, rare earth elements, amino acids,pharmaceuticals, pesticides etc. from wastewater solution or from their product source. Reactive extraction being clean process since the solvent system can be completely recovered and reuse, along with simple and cheap operation had created a lot of scope of it as an efficient recovery process. The primary hindrance in the establishment of the reactive extraction process for recovery of the acid is the search for an efficient and effective solvent.

1.2 Desirable Properties of Solute and Solvents

Addition of solvent in the aqueous carboxylic acid solution leads to form two phases where some carboxylic acid transfers from aqueous phase to organic phase. The extent of transfer depends on both the solute (carboxylic acid) and solvent properties.

1.2.1Properties of solute

In order to use reactive extraction process for removal of solute a certain desirable properties of it are recommended so that complexation reactions are favoured, such as:

A) Lewis acid or Lewis base functional groups: The solute of interest should have one or more functional group that can help to form moderately strong complex. Since most complexation process involve interaction of lewis acid with lewisbases, acidic and basic functional group are useful.

B) Low solute concentration: Since complexing agents provide particularly high equilibrium distribution for low solute concentration and tend to saturate stoichiometrically at high concentration,complexationis must attractive for relatively dilute aqueous solutions.

C) Low activity coefficients in water: The complexation separation processes are useful for hydrophilic solutes, so that phases should separate perfectly .

D) Low solute volatility: Solutes that are less volatile than water are good candidates for separation by complexation, so that while yhe time of extraction solute cannot be volatized

1.2.2Properties of solvents

In the process of extraction, separation is obtained by partition of solute between two immiscible phases. The two phases should have sufficient density difference and minimum surface tension so that there is a clear phase separation obtained once the phases are made to settle. The affinity of solvent to water should be very less and hence nonpolar solvents are preferred in the extraction process. The solute should have high solubility in the solvent. The solute should be easily recoverable from the solvent while conventional separation process is used.

1.3 Properties of Complex

In the process of reactive extraction the reaction between solute and extractant is the prime step. If the reaction produces a product with high bond energy then it will be difficult to obtain the solute back by some means of reversing the reaction economically. So the choice of extract is crucial here. It should form a complex with the solute where bond energy of the complex is low: less the 50kJ/mol and it is same to ordinary associations by vander-waals forces. A simple compexation reaction is

Solute + n. Extractant ⟷ Complex

The associated equilibrium constant is defined as

$$K_E = \frac{[COMPLEX]}{[SOLUTE]*[EXTRACTANT]^N}$$

In the above equation shows that the attractiveness of the process lies in removal of solute from relatively low solute concentration, since it could lead to higher complexation constant (K_E) . Another benefit of these complexation reactions is that these are selective for a certain group of solute, and hence, these processes have the potential to separate only certain solutes from complex mixtures. The complexeing reaction should have sufficiently fast kinetic in both direction so that equipment size should not become prohibitively large.

1.4 Distribution of Solute at Equilibrium state

The ratio of total concentration of solute in all its form (by partition dimmers and as complexes) in organic phase and total of all its existing form (dissociate and undissociated)in aqueous raffinate is used as the parameter called distribution coefficient to ascertain the success of a particular solvent.Mathematically it is

$$K_D = \frac{[A]\mathbf{org}}{[A]\mathbf{aq}}$$

Where, K_D is the distribution coefficient, $[A]_{org}$ and$[A]_{aq}$ are the concentration of solute A in organic and aqueous phases respectively.

1.5 Objective of Experiment

The present work was inspired by Amit [1]. They extracted propionic acid using tri-iso-octalaymine as extractant in solvents like toluene, petroleum ether, n Hexane. No work is yet reported to recover acetic acid from its aqueous solution using the same extractant in the same solvent. Thus, the extensive literature survey followed by isolation of grey areas leads to select the followings as the objective of the present work.

- Recovery of acetic acid using solvents like toluene, petroleum ether, n-hexane and turpentine oil.

- Enhancement of recovery of acetic acid using reactive extractant tri-iso-octylamine.

1.6 Organization of Thesis

Chapter 1 introduces the idea of reactive extraction along with proprieties of solvent, extractant and complexes. It also defines the objective of the present work.

Chapter 2 details with the past work related to basic reactive extraction of carboxylic acid and various parameters, which affect the extraction and kinetic studies especially on the recovery of acetic acid from the aqueous solution using the reactive extraction mechanism.

Chapter 3 includes the description of experimental set up and procedure. The chapter also contain the brief description of the solvent used.

Chapter 4 comprises the results of the present work. The Physical explanations of the results are also given.

Chapter 5 deals with the overall conclusion and future recommendation

CHAPTER 2

LITERATURE REVIEW

2.1 **Extraction by Amine**

Recovery of carboxylic acids from aqueous solutions and fermentation broths, where it is present in dilute form (<10%), is always of interest to researchers [2], [3], [4] and [5]. Reactive extraction depends on number of parameters like types of complexes, distribution coefficient, complexation-equilibrium constant, loading ratio, properties of the solvent etc. The amine based extractants and phosphorous based oxygen extractant are the most suitable. Aliphatic amines are efficient and comparatively low-cost extractants that have been used effectively to extract acetic acid [6].

Amine family divided as primary secondary tertiary and quaternary amines and theirbehaviour are

- Primary amines are soluble in water so it goes in aqueous phase.
- Secondary amines and Quaternary amines are more toxic for microorganisms. But it gives more distribution coefficient.[7]
- Tertiary amine extractant are effective and having high K_D value.[8]

2.1.1 Dependency of distributive coefficient (K_D)

It depends on the followings:

- Nature of the diluents used.
- Concentration of amine in those diluents.
- Carbon number in the tertiary amine which increases the extractability. It happens due to increase ofbasicity and polarity of amine with chain length.

The amine extractants are dissolved in diluent that dilutes the extractants to desired composition and controls the viscosity and density of solvent phase. It has been found that, in extraction of acid by amine, the type of diluents and the composition of solvent diluents mixture effect the equilibrium. Polar diluents have is more effective diluents than non polar ones due to higherpartition coefficients [9].

2.1.2 Complex formation

Wennersten[10] determined the structure of the acid amine complex in the diluents. He proposed that acid interacts with the amine directly to form an ion pair, and then [OH] of another molecule of acid forms a hydrogen bond with the conjugate [CO] of the carboxylate

of the first acid to form a complex. The diluted extractant gives much higher K_D values than the pure extractant [11].

2.2 Effect of Diluents

Liquid–liquid equilibria of different carboxylic acids using tri-Octayl Amine (TOA) in 1-octanol, chloroform, methyl isobutyl ketone (MIBK), tetra-chloromethane and hexane was investigated[12] to show the effect of acid and effect of diluents. The trend of strength of solvation and different model parameters based on mass action law was represented. Moralesset al. [13] studied the extraction equilibria for the systems tri-n-butylphosphate (TBP) and tri-n-octylamine (TOA) in organic diluents (decanol + dodecane) for five lower carboxylic acids. Gu et al. [14] studied the reactive extraction of propionic acid using Alamine 304-1 in 2-octanol, 1-dodecanol as diluents using various amine concentrations (0–100%), and found extraction to be maximum at the amine concentration between 20% and 40%

2.3 Distribution Coefficient

Earlier research has shown that phosphoryl and aliphatic amine is efficient extractant for carboxylic acid recovery [15], where extractant plays important role in complex formation. Also solvent affects the distribution coefficient. Physical property of solvent like viscosity and density affect the stability of complex [16].Forachieving high distribution coefficient solvent should have property like immiscible in water, and have higher value of dipole moment.

2.4 Equilibrium Studies on Reactive Extraction of Carboxylic Acids

Many researchers have tried to optimize the reactive extraction, also to predict the kinetic model of extraction [17], [18], [19], [10], and [21].They have considered the following parameters for optimization

- Concentration of carboxylic acid in aqueous phase
- Type of solvent
- Relative amount of solvent
- Type of extractant according to their bond nature
- Toxicity of solvent phase for micro-organismif applicable

Some researcher [22] tries to recover carboxylic acid from fermentation broth and try to optimize the recovery [23]

2.5 Different Extractant and Their Extraction Ability

Extractant are broadly divided as phosphoryl and tertiary amine compound. Wardell et al. [24] examined the effect of extractant on extraction of carboxylic acid. They have used phosphoryl extract: tri-butyl phosphate (TBP), di-butyl phosphate (DBP), tri-butyl phosphine oxide (TBPO), tri-phenylphosphineoxide (TPPO) and tertiary amine: tri-octyl amine (TOA), tri-iso-octyl amine (TIOA). The used solvents are chloroform, nitrobenzene, N-Hexanol, N-Heptanes. The highest extractability was obtained with high polarity of phosphoryl bond (P-O). The distribution coefficient of carboxylic acid was obtained in increasing order of phosphate < phosphonate < phosphinic oxide. The different capability indicates that with the increase in electronegativity, a decrease in the electron-donating ability and disappearance of Lewis basicity. In case of the amine it shows that there is no difference in extraction using tri-octyl amine and a tri-iso-octyl amine. However, these give higher distribution confident than tri butyl amine (TBA).

2.6 Enhancement of Extraction Process by Using Proper Solvent (Diluent)

The extract are generally viscous in nature, solvent can help it to improve the physical property The Diluent provides general solvation and affect the extraction power of the extractant by providing specific interaction. The diluent can also stop third phase formation that builds up due to the association of carboxylic acid and extractant [25].

Some diluents have active polar property and others are inert. Various active polar and proton donating diluents like - halogenated aliphatic/aromatic hydrocarbons, ketones, nitrobenzenes, higher alcohols enhance the extraction , The inert solvent like long chain paraffin, benzene restrict extraction capacity.

Mutual solubility of the phases was observed important in the extraction of carboxylic acids. The extent of this can be explained in terms of the nature and the total concentration of the component in the phases [26].

A few researchers mentioned that the stability of the ion pair formed is the determining factor in establishing the equilibrium conditions, Due to this reason, polar diluents were preferable to aliphatic and aromatic hydrocarbons [27] [28].

2.7 Effect of pH

Yang [29] studied the extraction of acetic, propionic, lactic and butyric acids with tertiary and quaternary amines at different pH values. It was noted that Aliquat 336, which is a quaternary amine salt, could extract both dissociated and undissociated forms of the acids. Hence, it was not affected by the pH of the aqueous solution like Alamine 336. Siebold [30] concluded that selection of the extractant should be based on the pH range of the aqueous solution of the acids and reusability of the extractant.

2.8 Effect of Temperature

San-Martin [31] has studied the recovery of lactic acid with tri-noctyl amine in xylene in the temperature range 293-323 K. They have investigated the effect of temperature on the loading and found that loading decreased with the increase in temperature.

Yang et al. [32] has proposed that with the formation of the complex, the system became more ordered and entropy decreased. Consequently, the amount of acid extracted decreased with the increase in temperature. It was concluded that the distribution coefficient of acetic acid in organic and aqueous phase decreases with increase in temperature, hence extraction of acetic acid decreases.

2.9 Solvent Toxicity in Reactive Extraction

Extractive fermentation was achieved successfully with tri-n-octyl methyl ammonium chloride (TOMAC) dissolved in oleyl alcohol. Gu [33] studied the extractive fermentation of propionic acid using Alamine 304-2 (tri-auryl amine) in 2-octanol, 1-dodecanol and Witcohol 85 NF (oleyl alcohol). Although 2-octanol yielded the highest distribution coefficients among the diluents studied, due to its toxicity to propionibacteria, it was not considered as the most appropriate solvent. Solvent toxicity was overcome by two strategies:

- By capturing the dissolved toxic solvent by vegetable oils.
- By using Witcohol 85 NF (oleyl alcohol) instead of 2-octanol.

Hong [34] found the optimum concentration of the extractant to achieve a high distribution coefficient in between 30 and 40% tri-n-octyl methyl ammonium.

CHAPTER 3

MATERIALS AND METHODS

A rigorous experimental work to extract carboxylic acid from the aqueous solution using reactive extraction method is carried out in the present work. The experiment is carried at atmosphearic pressure and room temperature in the range of 25-30^{o}C. It uses number of chemicals which are given in Table 3.1

Table 3.1: List of Chemicals used in the present work

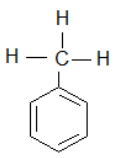
SN	NAME	Supplied by	Properties at 25 ^{0}C
1	Acetic acid	Merck specialties	Density 1.05g/ml, soluble in water
2	Toluene	Loba chemic	Density 0.886 g/ml, solubility in water 0.052%
3	N-hexane	Loba chemic	Density 0.658 gm/ml, solubility in water 0.01%
4	Turpentine oil	Local Market	Density 0.854-0.869 gm/ml, Insoluble in water
5	Petroleum ether	Merck specialties	Density 0.78 gm/ml, insoluble in water
6	Tri isooctalamine	Sigma aldrich	Density-0.816 g/ml, solubility in water 0.2g/lit
7	Phenolphthalein	Merck specialties	PH range- 8.2 to 10

3.1 Solvent Selection

In the present experiment different solvents like toluene, n-hexane, petroleum ether and turpentine oil are used to find best diluents out of these to extract acetic acid from aqueous solution.

The properties of these solvents are discussed below.

Toluene

The chemical structure of toluene is 

Toluene is widely used as solvent, because it has capability to dissolve organic and some inorganic chemical. as methyl group having electron donating nature due to that electron

density increases on benzene ring which create dipole moment for toluene . Similarly, it forms hydrogen bond with several inorganic chemical and work as a solvent for them.

n-Hexane

The chemical structure of n-Hexane is $H_3C- CH_2- CH_2- CH_2- CH_2- CH_3$.

n-hexane is aliphatic compound. It has very less dipole moment than toluene also its structure is uniform because of that there is very less probability of charge dislocation within the compound and hence, it is a non-polar solvent.

Petroleum ether

Petroleum ether is a group of volatile and highly flammable liquid hydrocarbon mixture and used as economic non-polar solvent.

Petroleum ether is obtained from petroleum refinery as the portion of the distillate which is intermediate between the lighter naphtha and the heavier kerosene. Its specific gravity is in between 0.6 to 0.8.

The following distillation fraction of petroleum ether is commonly available: 30 to 40^oC boiling point, 60 to 80^oC boiling point and 80 to 100^oC boiling point. The 60 to 80^oC boiling point petroleum ether fraction is used in the present work.

Turpentine oil

A thin volatile essential oil $C_{10}H_{16}$ is obtained by steam distillation or other means from the wood or excaudate of certain pine trees and used as paint thinner and solvent. It consist of aliphatic compound

3.2 Experiment Procedure

3.2.1 Preparation of stock solution

The synthetic aqueous acetic acid is prepared by mixing distilled water and glacial acetic acid. For the preparation of 0.1 gmol/lit of aqueous solution 5.76 ml of glacial acetic acid is added in 994.23 ml of distilled water with the help of micro pipette. Similarly, other aqueous acetic acid solution is prepared for a number experimental run.

3.2.2 Mixing of solvent and aqueous acetic acid solution

The solvent (Toluene, n-hexane, oil Petroleum ether or Turpentine) and aqueous solution are immiscible to each other. Hence, the mixture is shaken for 12 hours in an incubator shaker with 120 rpm. Thus both the phases get intermixed.

After mixing, sample should be kept for 2 hours for the exact separation of two phases, as separation over the aqueous phase settle down at the bottom part of solution and organic on upper part, for titration take sample from aqueous part using pipette.

3.3 Sampling and Titration

3.3.1 Preparation of alkali solution

For alkali titration use sodium hydroxide (NaOH) as known component, for the preparation of alkali solution (NaOH) having 0.02 gmol/lit concentrations add 0.8 gm of pure NaOH in 1 litter of distilled water. As the NaOH is hygroscopic in nature so there may be mistake on weight measurement so for standardization of alkali solution using potassium hydrogen phthalate (KHP).

3.3.2 Estimation of acetic acid in aqueous phase

It is estimated experimentally titrating the solution with caustic solution. Phenolphthalein is used as indicator. The data is recorded when solution becomes pink in colour. The titration is repeated three times for the same sample and average reading was taken for estimating acetic acid.

Normality is defined as number of gram equivalents of the solute present in 1000 ml solution. If W g of solute of equivalent weight E is present in V mL of the solution, the normality of the solution is given by

$$\text{Normality} = \frac{W*100}{E*V}$$

In the neutral solution,

$$\mathbf{N_1V_1=N_2V_2}$$

Where N_1 normality of titrant, aqueous solution of acetic acid

N_2 normality of titrate, caustic solution

V_1 volume of titrant, aqueous solution of acetic acid

V_2 volume of titrate, caustic solution

N_1 is calculated for the know values of V_1, N_2 and V_2.

3.4 Over-view of the Experiment Procedure

Complete overview is shown in Fig. 3.1. It starts from preparation of standard solution and then ends with complete analysis along with overview of intermediate procedure.

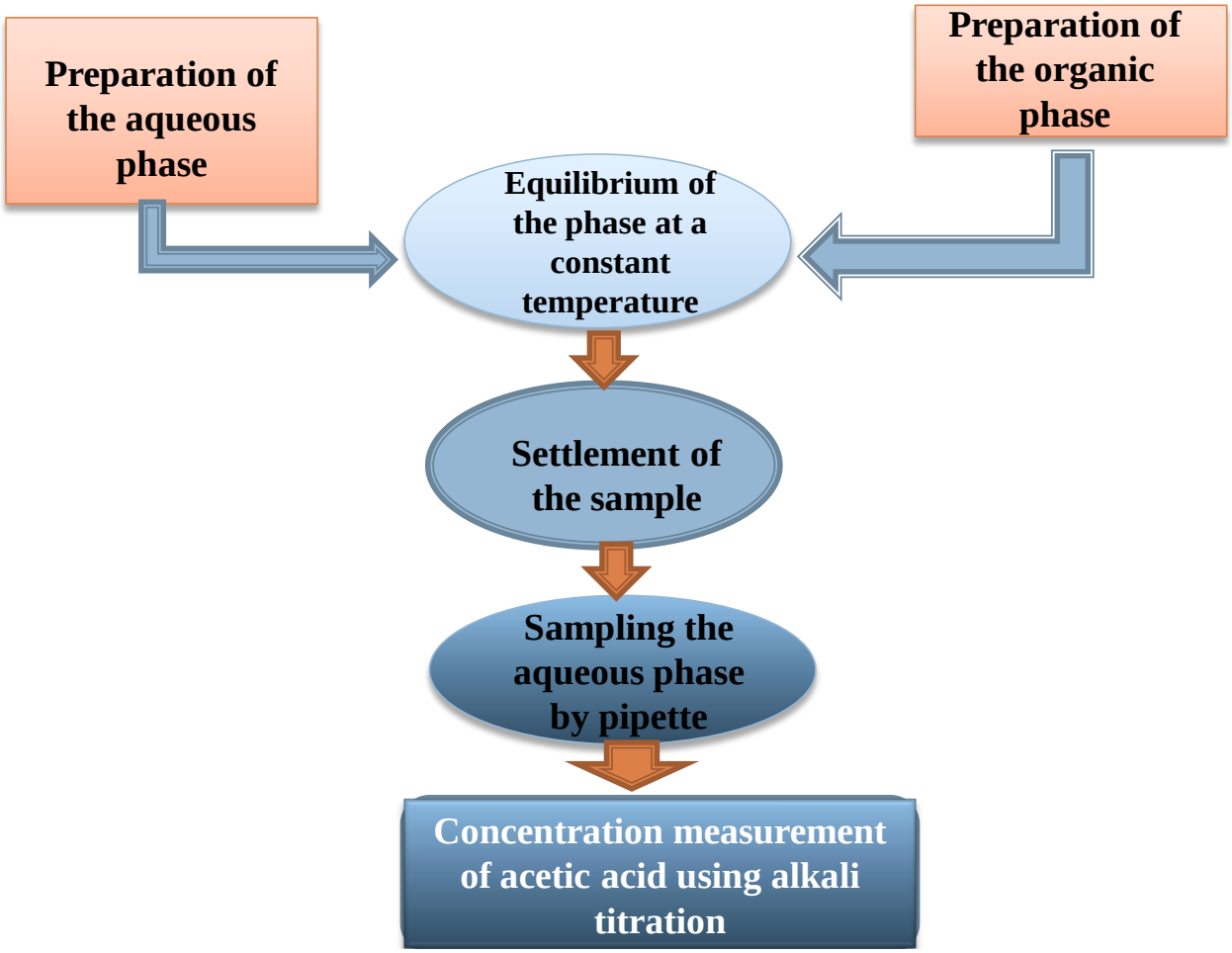


Figure 3.1: Overview of procedures

CHAPTER 4

RESULTS AND DISCUSSIONS

4.1 Extraction of Acetic Acid Using Only Solvents

The distribution of acetic acid both in the aqueous and organic phases starting with different acetic acid concentration in the aqueous phase is studied and the data are given in Table 4.1.

Table 4.1 Distribution of acetic acid in different organic solvent

Initial [A] concentration	Toluene		Petroleum ether		n-Hexane		Turpentine oil	
Aqueous solution gmol/l	[A]aq gmol/l	[A]org gmol/l	[A]aq gmol/l	[A]org gmol/l	[A]aq gmol/l	[A]org gmol/l	[A]aq gmol/l	[A]org gmol/l
0.1	0.058	0.041	0.066	0.033	0.076	0.023	0.064	0.035
0.2	0.105	0.094	0.125	0.075	0.142	0.057	0.0121	0.078
0.3	0.142	0.157	0.176	0.123	20.00	0.100	0.171	0.128
0.4	0.173	0.226	0.222	0.177	0.250	0.150	0.216	0.183
0.5	0.200	0.300	0.263	0.236	0.294	0.205	0.256	0.243

The tabulated data shows different distribution pattern of acetic acid in different solvents and toluene is found as the best solvent in the present study. Acetic acid interacts more with the solvents having higher dipole moments. The order of dipole moments of the used solvents are toluene > petroleum ether ≈ turpentine oil > n-hexane. Thus the distribution of acetic acid given in Table 4.1 is justified.

Graphical distribution of acetic acid in aqueous and organic phases is shown in Fig. 4.1. It shows that the concentration of acetic acid in solvent increases with the increase in acetic acid concentration in the aqueous phase.

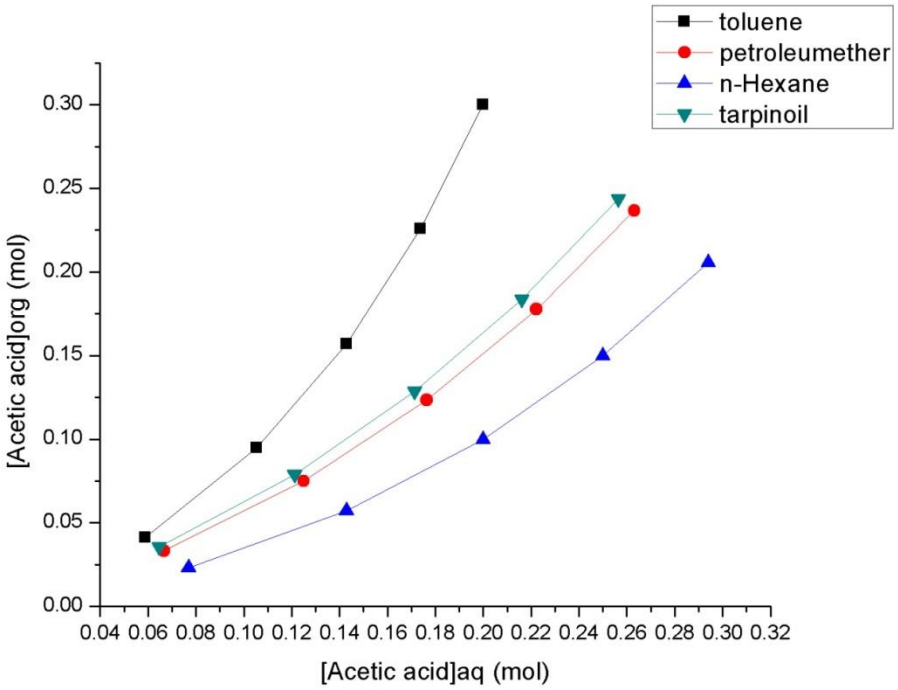


Figure 4.1: Acetic acid distribution in aqueous and organic phase in different solvent using same initial concentration of acetic acid.

The distribution coefficient as already defined in chapter 2 is $K_D = \frac{[A]\mathbf{org}}{[A]\mathbf{aq}}$

Against the experimental data of Table 4.1 the calculated distribution coefficients are given in Table 4.2.

Table 4.2 Distribution coefficient of solvent with different concentration of aqueous solution

Concentration of aqueous solution initially used (gmol/l)	Distribution coefficient in different solvent			
	Toluene	Petroleum ether	N-Hexane	Turpentine oil
0.1	0.70	0.54	0.29	0.50
0.2	0.90	0.64	0.39	0.60

0.3	1.10	0.74	0.50	0.70
0.4	1.29	0.85	0.60	0.79
0.5	1.50	0.94	0.70	0.90

The tabulated data show that the distribution coefficients values are less than 1.0 except toluene with high initial acetic acid concentration. It occurs due more affinity of acetic acid towards polar water than the organic solvents.

The variation of distribution coefficients with the initial acetic concentration for different solvents is depicted in Fig. 4.2. It shows a linear increasing trend of distribution coefficient with the initial concentration of acetic acid for all solvents. The rate of increase is found maximum for toluene.

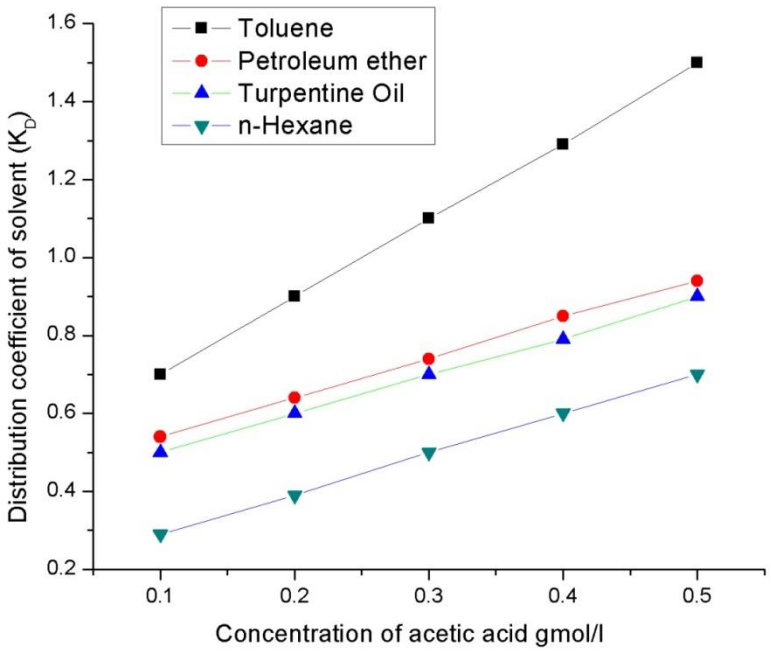


Figure 4.2: The variation of distribution coefficients with the initial acetic concentration for different solvents.

4.2 Extraction of Aqueous Acetic Acid (0.2gmol/l) Using tri-iso-octylamine (Extractant) in Solvents

The distribution of acetic acid in both aqueous and organic phases with different amount of tri-iso-octalymaine in solvent is tabulated in Table 4.3.The initial concentration of acetic acid is kept same (0.2 gmol/l) in all the experimental runs.

Table 4.3 Distribution of acetic acid in different organic solvent with different amount of tri-iso-octylamine in diluents. The initial acetic acid concentration is 0.2 gmol/l.

Extractant % by volume in solvent	Toluene		Petroleum ether		n-Hexane	
Aqueous solution (0.2 gmol/l)	[A]org gmol/l	[A]aq gmol/l	[A]org gmol/l	[A]aq gmol/l	[A]org gmol/l	[A]aq gmol/l
10	0.092	0.108	0.078	0.122	0.080	0.120
20	0.126	0.0740	0.111	0.090	0.1130	0.087
30	0.144	0.0560	0.133	0.0670	0.134	0.066
40	0.157	0.0430	0.146	0.054	0.145	0.055

Table 4.3 shows different distribution pattern of acetic acid in different solvents with different percentage (by volume) of tri-iso-octylamine in solvent. The comparison of the data of Table 4.3 with Table 4.1 depicts that there is an intensification of acetic acid transfer to organic phase in presence of extractant. This increase is due to the formation of complex of acetic acid with tri-iso-octyl amine in the organic phase. The related distribution coefficient values are shown in Table 4.4. The data shows a large increase of distribution coefficient in presence of extractant for all three selected solvents. The increase is around 300% at larger volume fraction of reactive extractant. Therefore, it can be concluded that the reactive extraction favours the transfer the acetic acid to solvent.

Table 4.4: Distribution cofficent of acetic acid with different amount of tri-iso-octylamine in diluents. The initial acetic acid concentration is 0.2 gmol/l.

Amount of tri-isooctylamine used in solvent (% by volume)	Distribution coefficient in different solvent		
	Toluene	Petroleum ether	N-Hexane
10	0.76	0.75	0.59
20	1.21	1.22	0.96
30	2.30	1.87	1.80
40	3.34	2.60	2.30

4.3 Extraction of Aqueous Acetic Acid (0.4gmol/l) using tri-iso-octylamine (Extractant) in Solvents

Similar experiments are carried out with initial acetic acid concentrations of 0.4 glom/lit. The equilibrium distributions of acetic acid in both the aqueous and organic phases are given in Table 4.5.

Table 4.5: Distribution of acetic acid in different organic solvent with different amount of tri-iso-octylamine in diluents. The initial acetic feed concentration is 0.4 gmol/l.

Extractant % by volume in solvent	Toluene		Petroleum ether		n-Hexane	
	[A]org gmol/l	[A]aq gmol/l	[A]org gmol/l	[A]aq gmol/l	[A]org gmol/l	[A]aq gmol/l
10	0.149	0.251	0.143	0.257	0.174	0.226
20	0.245	0.255	0.220	0.180	0.220	0.181
30	0.286	0.114	0.261	0.139	0.320	0.140
40	0.308	0.092	0.289	0.111	0.279	0.121

The comparison of data of table 4.4 with 4.5 depicts that there is an increase in acetic acid in organic phase. Therefore it can be concluded that more amount of acetic acid extracted if concentration of it in aqueous solution is more.

The computed distribution coefficient from Table 4.5 is shown in Table 4.6. The initial acetic concentration has some positive effect on the distribution coefficient. All the distribution coefficients increase by 10-15% due to increase in acetic acid feed concentration from 0.2 to 0.4 gmol/l. The relative comparison is also made in Table 4.7 and Fig. 4.3.

Table 4.6: Distribution coefficient of acetic acid in different organic solvent with different amount of tri-iso-octylamine in diluents. The initial acetic feed concentration is 0.4 gmol/l.

Amount of tri-isooctylamine used in solvent (% by volume) In 0.4 gmol/lit of aqueous solution	Distribution coefficient in different solvent		
	Toluene	Petroleum ether	N-Hexane
10	0.85	0.66	0.63
20	1.70	1.29	1.23
30	2.57	2.03	1.98
40	2.65	2.70	2.63

Table 4.7: Distribution cofficent of acetic acid in presence and absence of tri-iso-octylamine

Solvent name	Without extractant		With Extractant Initial aqueous phase concentration is 0.2 glom/lit				With Extractant Initial aqueous phase concentration is 0.4 glom/lit			
	0.2 glom/lit of initial aqueous solution	0.4 glom/lit of initial aqueous solution	Volume % of tri-iso-octylamine in solvent				Volume % of tri-iso-octylamine in solvent			
			10%	20%	30%	40%	10%	20%	30%	40%
Toluene	0.90	1.29	0.76	1.23	2.35	3.34	0.85	1.70	2.57	3.65
Petroleum ether	0.60	0.79	0.66	1.29	2.03	2.74	0.75	1.21	2.02	2.42
n-Hexane	0.39	0.60	0.59	0.96	1.80	2.30	0.63	1.23	1.98	2.63

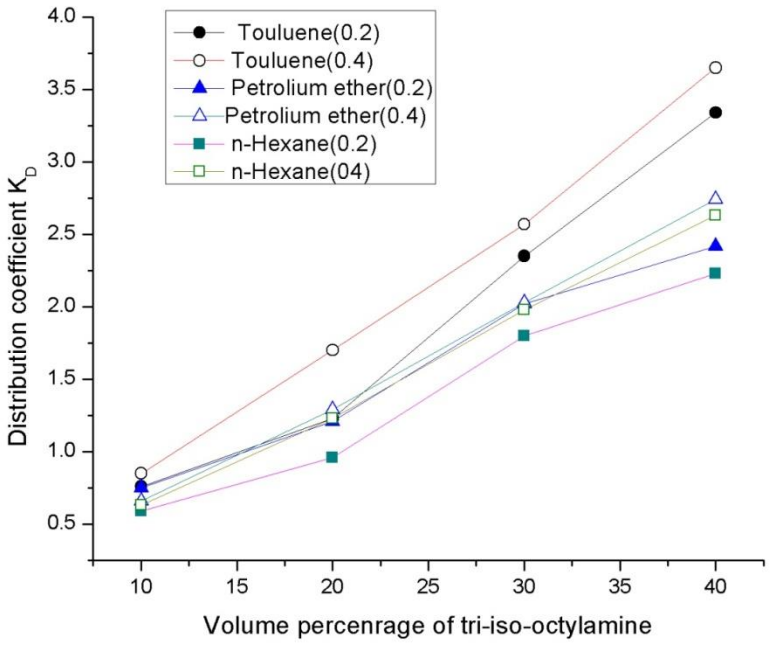


Figure 4.3: The variation of distribution coefficients with the volume percent of tri-iso-octylamin in solvent. The initial feed concentrations of acetic acid are 0.2 and 0.4 gmol/l.

CHAPTER 5

CONCLUSIONS & FUTURE WORK

The reactive extraction studies lead us to draw the following important conclusions:

- Acetic acid was extracted from the aqueous solution by reactive extraction process using tri-iso-octylamine as extractant in different organic solvents (toluene, petroleum ether, n-hexane).

- The distribution coefficients of acetic acid while extracted by only solvent had lower values of distribution coefficients. The values are 1.29, 0.79 and 0.6 for toluene, petroleum ether and n-hexane respectively while the initial acetic acid feed was kept 0.4 gmol/l.
- Enhanced distribution coefficients were obtained while acetic acid was extracted by reactive extraction process using tri-iso-octylamine as extractant.
- A 300% increase of distribution coefficient was obtained in case of reactive absorption process compared to the conventional absorption process.
- The increase of acetic acid concentration in the feed had increased the distribution coefficient of acetic acid by 10-15%.

The future work includes:

- In case of reactive absorption process, more experiments are required to carry out using a few more number of initial feed concentrations of acetic acid.
- Kinetic study can be carried out to find dynamic behaviour of reactive absorption process.

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